

L1 ANSWER 1 OF 2 CA COPYRIGHT 2002 ACS  
ACCESSION NUMBER: 71:13963 CA  
TITLE: Continuous Alfin catalyst polymerization of olefins  
INVENTOR(S): Baba, Theodore B.  
PATENT ASSIGNEE(S): National Distillers and Chemical Corp.  
SOURCE: Ger., Offen., 132 pp.  
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PATENT INFORMATION:

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			US	19680626

AB Alfin polymers are prep'd. by continuously mixing unsatd. org. monomers, Alfin catalysts, mol.-wt. regulators, and solvents and then carrying out the polymn. at elevated temps., regulating mol. wt. by the addn. of mol.-wt. moderators. The reaction mixt. is then treated with CO<sub>2</sub>, O, or an org. ester, with the CO<sub>2</sub> converting organometallic compds. into org. acid salts, the O converting organometallic compds. into the corresponding acetylenes and cyclopentadienes, and the org. esters converting acetylides and cyclopentadiene-organometallic compds. into metal salts, substituted acetylenes, and substituted cyclopentadienes. Liq. materials, unconverted monomers, liq. low polymers, and solvents are sepd. from the reaction mixt., water-sol. salts are extd., and the resulting dispersion is steam distd. and the solids recovered. The Alfin polymer is then washed and dried. Thus, a dispersion of 88 kg. liq. Na at 116.degree. in 231 kg. iso-octane (I) was prep'd. at 35 psig. A 231-kg. portion of this dispersion was mixed with 953 kg. I, and 54.4 kg. iso-PrOH was then added over 3 hrs. with cooling to hold the temp. at .apprx.66.degree.. BuCl (86.2 kg.) was then added over 5 hrs., the mixt. was stirred 1 hr., and 43.1 kg. propylene was added to convert the BuNa to allylsodium, giving butane as a by-product. This catalyst slurry (59 kg./hr.) was added to a reactor, where butadiene (II) and isoprene (III) were added at 218 and 54.4 kg./hr., resp., together with 1.59 kg./hr. 1,4-dihydronaphthalene mol.-wt. regulator and 1934.5 kg./hr. I solvent. All addns. were made at 38.degree.. The reaction temp. was held at 66-72.degree. by cooling, and the pressure was 50 psig. The reaction mixt. was fed through a series of reactors, over a total reaction time of .apprx.3 hrs. The polymer-contg. effluent was then passed at the rate of 56.8 l./min. into a carboxylation app., where it was treated with 878 l./hr. CO<sub>2</sub>. Residence time in this

reactor was 1 hr. The carboxylated mixt. was passed at 2227 kg./hr.

into

a solvent stripper, where it was mixed with water at 88.degree. to ext. water-sol. salts, heated to 100.degree. by the addn. of steam and hydrocarbon vapors, and distd. to remove solvents and residual monomers. I, II, and III were sepd., dried, and recycled. II dimer was sepd. in a column for heavy fraction and discarded. The treatment of the polymer-contg. effluent with O or PhOAc was carried out similarly. The product was obtained in the form of a rubber crumb contg. .apprx.60 wt.

%

water and only small amts. of mol.-wt. regulator and solvent. The crumb was washed with 49.2 l./min. of cold water, pressed to reduce the water content to .apprx.9%, heated to .apprx.149.degree. under pressure, and placed in an expansion chamber, where steam and solvent were boiled off. The product was then ready for processing. This process is economical, since solvents and unreacted raw materials are recycled. The use of II alone and in mixts. with styrene as monomers was also claimed.

L1 ANSWER 2 OF 2 WPIX (C) 2002 THOMSON DERWENT  
ACCESSION NUMBER: 1968-24330Q [00] WPIX  
TITLE: Purification step in a continuous alfin polymerisation.  
DERWENT CLASS: A00  
PATENT ASSIGNEE(S): (NADI) NAT DISTILLERS & CHEM CORP  
COUNTRY COUNT: 7  
PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG
BE 721854	A	(196800)*			
DE 1800935	A	(196801)		<--	
NL 6814276	A	(196801)			
CA 833658	A	(197006)			
FR 1589522	A	(197031)			
JP 46034986	B	(197140)			
US 3640980	A	(197211)			
CA 912907	A	(197245)			

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AB BE 721854 A UPAB: 19930831

A continuous alfin polymerisation process includes a step whereby after the finish of polymerisation the reaction mixture is treated with one of carbon dioxide, oxygen or an organic ester to react with the acetylide and metal derivatives of cyclopentadiene which are present in the reaction mixture.

The recycling of solvent containing increasing amounts of cyclopentadiene and acetylene would have poisoned the catalyst.

The ester is the ester of an alkyl alcohol and an aliphatic acid which contains 1-20C. The ester is of an alkyl or aromatic alcohol and an aliphatic or aromatic acid containing 6-20C.